BRITISH STANDARD

BS 4267-2:
1988

Ammonium nitrate —

Part 2: Method for determination of total nitrogen content

NOTE It is recommended that this Part of BS 4267 read in conjunction with the information given in the "General introduction", published separately as BS 4267-0.

WARNING. Ammonium nitrate is a strong oxidizing agent. If necessary, break the test sample up, by crushing rather than grinding.

UDC 661.525:546.39:549.751.13:543:620.1

Foreword

This Part of BS 4267 has been prepared under the direction of the Chemicals Standards Committee. It supersedes clause **3** of BS 4267:1968, which is withdrawn.

This Part of BS 4267 is related to BS 5551-4.1.3:1986 which is identical with ISO 5315:1984.

This British Standard describes a method of test only, and should not be used or quoted as a specification defining limits of purity. Reference to this Part should indicate that the method of test used is in accordance with BS 4267-2:1988.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on 29 July 1988

The Committees responsible for this British Standard are shown in Part 0.
The following BSI references relate to the work on this standard:
Committee reference CIC/21
Draft for comment 84/54890 DC

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Part of BS 4267 describes a titrimetric method for the determination of the total nitrogen content of ammonium nitrate for industrial use.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

The nitrate nitrogen in a sample is reduced to ammonia by chromium powder in an acid medium. Ammonia is distilled from an alkaline solution, absorbed in an excess of standard volumetric sulphuric acid solution which is back-titrated with standard volumetric sodium hydroxide solution, with methyl red or screened methyl red as indicator.

3 Reagents

3.1 *General*. During the analysis, use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978.

3.2 *Chromium metal*, powder, of particle size not greater than 250 μm.

3.3 *Ammonium nitrate*, anhydrous. Heat approximately 5 g of ammonium nitrate on a clean platinum dish, in an oven controlled at 100 ± 2 °C, cool in a desiccator and weigh. Repeat this procedure until successive weighings do not differ by more than 0.01 g.

3.4 *Hydrochloric acid solution*, ρ = approximately 1.18 g/mL.

3.5 *Sodium hydroxide solution*, approximately 400 g/L.

3.6 *Sodium hydroxide, standard volumetric solution*, c(NaOH) = 0.10 mol/L.

3.7 *Sulphuric acid, standard volumetric solution*, $c(H_2SO_4)$ = 0.10 mol/L.

3.8 *Sulphuric acid, standard volumetric solution,* $c(H_2SO_4)$ = 0.05 mol/L.

3.9 *Indicator solution*, either a) or b).

a) *Screened methyl red indicator solution*. Mix 50 mL of a 2 g/L solution of methyl red in 95 % (*V*/*V*) ethanol with 50 mL of a 1 g/L solution of methylene blue in 95 % (*V*/*V*) ethanol.

b) *Methyl red indicator solution*. Dissolve 0.1 g of methyl red in 50 mL of 95 % (*V*/*V*) ethanol.

NOTE For the purposes of **3.9**, the ethanol may be replaced by industrial methylated spirits 95 % (*V*/*V*) complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquors Duties Act 1972, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

3.10 *pH indicator paper*, wide range.

4 Apparatus

4.1 *Ordinary laboratory apparatus*

4.2 *Distillation apparatus* (see Figure 1)

4.2.1 *General*. The components of the distillation apparatus shall be connected by means of rubber bungs and tubing, or by the use of spherical ground glass joints.

NOTE Rubber bungs and tubing should be replaced when they begin to perish or show signs of wear.

The apparatus comprises the items listed in **4.2.2** to **4.2.5**.

4.2.2 *Flask*, either a round-bottomed flask, 1 000 mL, or a Kjeldahl flask, 800 mL.

4.2.3 *Single-bulb splash head and separate open-top dropping funnel*, 100 mL, followed by a delivery tube at the outlet.

NOTE Information on splash heads is given in BS 6855.

4.2.4 *Allihn condenser*, seven-bulb, with an expansion bulb of capacity approximately 100 mL, followed by a delivery tube at the outlet (see BS 5922).

4.2.5 *Receiver*, either a conical flask or beaker, 500 mL.

4.3 *Burettes*, class A, 50 mL, complying with BS 846.

4.4 *One-mark volumetric flask*, class A, 500 mL, complying with BS 1792.

4.5 *Glass beads*, 2 mm to 3 mm in diameter.

5 Procedure

5.1 Test portion and preparation of the test solution

Weigh 5.000 g of the test sample and transfer to the one-mark volumetric flask (**4.4**). Dissolve in water, dilute to the mark and mix well. Filter through a dry filter paper, discarding the first 50 mL of filtrate.

5.2 Determination

5.2.1 *Reduction*. Transfer 25.0 mL of the test solution (**5.1**) to the flask (**4.2.2**) and add sufficient water to make up the total volume to 35 mL.

Add 1.2 g of the chromium powder (**3.2**) and 7 mL of the hydrochloric acid solution (**3.4**). Allow the flask to stand for at least 5 min, but not more than 10 min, at ambient temperature.

Place the flask on a heating device in a fume cupboard with the heat input regulated to a level at which 250 mL of water at 20 °C would be brought to a "rolling" boil in 7.0 min to 7.5 min. Heat the flask for 4.5 min. Remove from the heat and allow to cool.

5.2.2 *Distillation*. Add about 250 mL of water to the flask (**4.2.2**), and a few of the glass beads (**4.5**). Assemble the apparatus as shown in Figure 1.

By means of a burette (**4.3**) measure into the receiver (**4.2.5**) 50.0 mL of the 0.10 mol/L standard volumetric sulphuric acid solution (**3.7**). Add four or five drops of the indicator solution [**3.9** a) or b)] and place the receiver so that the end of the delivery tube (**4.2.4**) is below the surface of the acid, adding water to the receiver if necessary.

Pour 30 mL of the sodium hydroxide solution (**3.5**) into the dropping funnel (**4.2.3**), and carefully run all but about 2 mL of this solution into the flask (**4.2.2**). Close the stop-cock, leaving the remaining 2 mL in the dropping funnel. Bring the contents of the flask to the boil, increasing the rate of heating progressively until the contents of the flask are boiling briskly.

When at least 150 mL of distillate has collected, partially withdraw the receiver so that the delivery tube rests on the rim of the receiver. Test the subsequent distillate with the pH indicator paper (**3.10**) to ensure that all the ammonia has completely distilled. Remove the source of heat.

Detach the splash head from the condenser (**4.2.4**) and wash the insides of the condenser and expansion bulb with water, collecting the washings in the receiver. Rinse the outside of the delivery tube and collect the rinsings in the receiver.

5.2.2 *Titration*. Back-titrate the excess of acid with the standard volumetric sodium hydroxide solution (**3.6**) to the neutral colour of the indicator.

5.3 Blank test

Carry out a blank test at the same time as the determination, using the same reagents but omitting the test solution (**5.1**), and using the 0.05 mol/L standard volumetric sulphuric acid solution (**3.8**) instead of the 0.10 mol/L standard volumetric sulphuric acid solution (**3.7**).

The result of the blank test should not exceed 1.0 mL of the 0.05 mol/L standard volumetric sulphuric acid solution (**3.8**). If the result is greater than 1.0 mL, check the reagents, especially the chromium powder (**3.2**), by repeating the procedure described in clause **5**, using fresh reagents as necessary.

5.4 Check test

Carry out a periodic check on the efficiency of the apparatus and the accuracy of the method using an aliquot portion of a freshly prepared solution of the ammonium nitrate (**3.3**) containing 0.1 g of nitrogen. The check shall be made under the same conditions as the determination and the blank test and using the same indicator.

6 Expression of results

The total nitrogen content, expressed as a percentage by mass of nitrogen (N), is given by the expression

$$\frac{[(2V_1 - V_2) - (V_3 - V_4)] \times 14.01 \times 0.1 \times V_5 \times 100}{1000 \times V_6 \times m}$$

where

V_1 is the volume of the 0.10 mol/L standard volumetric sulphuric acid solution (**3.7**) used for the determination (in mL), i.e. 50 mL;

V_2 is the volume of the sodium hydroxide solution (**3.6**) used for the determination (in mL);

V_3 is the volume of the 0.05 mol/L standard volumetric sulphuric acid solution (**3.8**) used for the blank test (in mL), i.e. 50 mL;

V_4 is the volume of the sodium hydroxide solution (**3.6**) used for the blank test (in mL);

V_5 is the volume of the test solution before filtering (see **5.1**) (in mL), i.e. 500 mL;

V_6 is the volume of the aliquot portion taken from the test solution (see **5.2.1**) (in mL), i.e. 25 mL;

m is the mass of the test portion (**5.1**) (in g), i.e. 5.000 g.

This expression reduces to the following.

$[(V_4 - V_2) + 50] \times 0.56$

7 Test report

The test report shall include the following information:

a) a complete identification of the sample;

b) a reference to this British Standard, i.e. BS 4267-2:1988;

c) the results expressed in accordance with clause **6**;

d) any unusual features noted during the determination;

e) any operation not included in this Part of BS 4267 or regarded as optional.

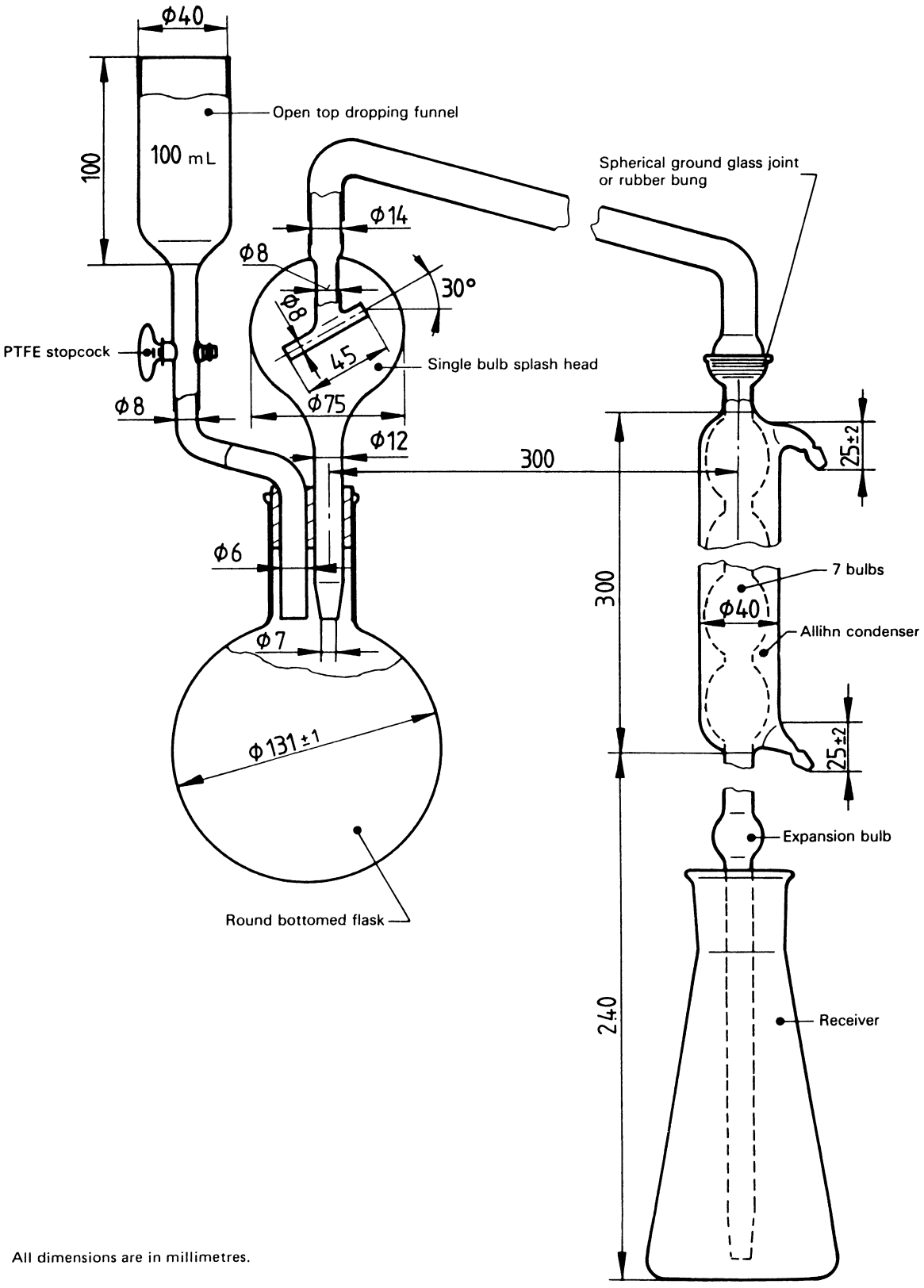

Figure 1 — Typical distillation apparatus (using a round bottomed flask)

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Publications referred to

BS 846, *Specification for burettes.*
BS 1792, *Specification for one-mark volumetric flasks.*
BS 3591, *Specification for industrial methylated spirits.*
BS 3978, *Specification for water for laboratory use.*
BS 4267, *Ammonium nitrate.*
BS 4267-0, *General introduction.*
BS 5551, *Fertilizers.*
BS 5551-4.1.3, *Determination of total nitrogen content (titrimetric method after distillation)*[1)].
BS 5922, *Specification for glass condensers for laboratory use.*
BS 6855, *Recommendation for design and construction of glass splash heads for laboratory use.*
ISO 5315, *Fertilizers — Determination of total nitrogen content — Titrimetric method after distillation*[1)].

[1)] Referred to the forword only.

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London
W4 4AL